RESEARCH ARTICLE

Synthesis, Characterization and Application of Talc/Fe_3O_4 Nano Composite in the Photocatalytic Degradation of Methylene Blue Dye

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ABSTRACT: The degradation of Methylene Blue dye using Talc/Fe_3O_4 nano composite material was studied. Talc/Fe_3O_4 nano composite material was successfully synthesized by co-precipitation method. The SEM, XRD,FT-IR,EDX studies were carried out for characterization of Talc/Fe_3O_4 Nano composite material. The XRD and SEM morphology indicates the Nano composite material has crystalline size 49.1nm and circular shape at higher magnifications respectively. The FT-IR showed the bonds between functional groups and FeO group, Si-O bending and vibration bonds. The presence of FeO, Fe, C, in nano composite material were confirmed by EDX .The Photo catalytic degradation studies were carried out for water soluble methylene blue (MB) dye by using Talc/Fe_3O_4 nano composite in aqueous solution. Different parameters like dye concentration, dosage of photo catalyst, contact time and pH were studied. It was observed that photo catalytic degradation by Talc/Fe_3O_4 nano composite is more effective. 87% degradation of MB dye at initial concentration 5ppm, photo catalytic dose 30mg, pH 9, and contact time of 150 min was observed.

KEYWORDS: Talc/Fe_3O_4 nanocomposites, XRD, SEM, Methylene Blue dye, UV-Vis Spectrophotometry, Photocatalytic reactor.

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1. INTRODUCTION

Nanotechnology fields a hot field in which design and synthesis of nanometer-scaled structures [1-2]. NPs research has become an area of attention due to their unparalleled superior properties compared to their bulk materials**,** which results in their wide range of applications in dissimilar fields [2-3]. Their high surface energy with a high surface area to volume ratio and relatively small sizes exhibits favorable properties like catalytic properties, thermal properties, electrical conductivity, optical properties, and biological applications [4, 5]. Fe_3O_4 particles demonstrates fascinating magnetic behavior, owing to unique potential applications in versatile fields, like catalysts, medical diagnosis, ferro-fluids, high-density magnetic recording media [6,7]. Magnetic nano-particles has appealed substantial interest in biomedicine and biomedical engineering for applications, admitting magnetic carriers for drug delivery and magnetic resonance imaging (MRI)[8]. Magnetic nano-particles posses super paramagnetic characteristics with higher saturation magnetization, and good biocompatibility behavior prompted researchers for the economic expenditure. Fe_3O_4 is one of the preferred well-known filler materials, which is combined with polymers/nano-composites to be used as magnetic recording media, and in medical applications [9]. Number of research works confirmed the iron oxides including γ-Fe_3O_4 (magnetite), Fe_3O_4, and α-Fe_3O_4 (hematite) particles; from these Fe_3O_4 is a very anticipating candidate as its biocompatibility. Talc is a clay mineral of hydrated form of magnesium silicate ($Mg_3Si_4O_{10}$ $(OH)_2$). The talc surface is extremely hydrophobic and, thus, in a finely ground state and float on the surface of water. The softness of talc is due to the easy displacement of these layers, rendering the material a poor electrical and thermal, refractory behavior, and resistant to acids [10]. It is broadly used in industrial products such as ceramics, putties, paper, paints, flame and ignition retardants, and active filler in polymer and rubber. The structure of talc is the well-known 2:1 (T–O–T) layer configuration consisting of a octahedral magnesium (Mg) coordinated sheet (O) sandwiched between two tetrahedral silicon (Si) coordinated sheets (T) [4]. Clothing, textiles, and agriculture, is the basic requirement of human being. High concentrations of textile dyes in water bodies stop the reoxygenation capacity of the receiving water and cutoffsunlight, thereby upsetting biological activity in aquatic life and also the photosynthesis process of

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aquatic plants or algae [11]

Dyes are a group of complex organic materials which enter the environment due to various processes like dyeing in textile industry. Dyes are classified into different types like Vat, Reactive, Direct, Cationic, Acidic, and Disperse etc. Textile industries produce wastewater with different chemical quality and quantity due to diversity of consumed dyes and production methods. In these industries, a huge amount of colored wastewater is produced that is usually poisonous, resistant to biodegradation and sustainable in environment, thus common biological methods aren't effective for removing almost all synthetic dyes due to complex chain structure and dye sustainable nature. Studies have shown that textile wastewater has low BOD/COD ratio (0.1) which is resulted from non-biodegradability of dyes. Discharge of colored wastewater resulted from textile industries to receiving waters will result in reduced penetration of sunlight, occurrence of eutrophication and interference with receiving water ecology, in addition to decreased photosynthesis of aquatic plants and algae in aqueous environments. To remove dyes from wastewater Physical, Chemical, Biological or advanced waste water treatment methods can be used [12].

The main aim of this work is to synthesis the Talc/Fe_3O_4 nano composite materials was successfully synthesized by co-precipitation method and understand the degradation of methylene blue dye using Talc/Fe_3O_4 nano composite. The XRD, SEM, FT-IR and EDX studies were carried out for characterization of Talc/Fe_3O_4 Nano composite material.

2. MATERIAL AND METHODOLOGY

2.1 Chemicals Used

Ferric nitrate hexahydrate ($FeCl_3 \cdot 6H_2O$), Ferrous chloride tetrahydrate($FeCl_2 \cdot 4H_2O$),Talcum powder ($Mg_3Si_4O_{10}(OH)_2$),Sodium hydroxide (NaOH),Methylene Blue. All aqueous solutions were prepared with deionized water [10].

2.2 Instruments Used

The pH meter is used for checking pH of solution whether acidic or base. Photocatalytic reactor is used photo reduction and degradation using light as a source. The UV-visible spectrophotometer is used to know the absorbance of the samples. X-Ray Powder Diffraction Method, Fourier Transform Infrared Resonance (FT-IR), Scanning Electron Microscope (SEM), Energy Dispersive X-ray Spectroscopy (E-DAX) were used for the characterization.

2.3. Experimental Procedure

The chemical co-precipitation technique has been used in preparation of Talc/Fe_3O_4 nano composite materials. 2.0 g of Talc was suspended in 120 ml deionised water, and then a solution of Ferric nitrate[$Fe(NO_3)_3 \cdot 9H_2O$] containing Fe^{3+} and ferrous chloride ($FeCl_2.4H_2O$) containing Fe^{2+} with (2:1) molar ratio was added into the mixture. The ion solution suspended with talc composites were stirred for 24 h for impregnation on the external surface of talc layers to prepare Talc/Fe^{3+}–Fe^{2+} composites. Then 25 ml of freshly prepared NaOH (2.0 M) was added to Talc/Fe^{3+}–Fe^{2+}composites suspension under continuous stirring until the mixture start precipitating at an ambient temperature. The suspensions were finally centrifuged at 4000 rpm, washed twice with ethanol and distilled water to remove impurities. The settled solids were taken in watch glass kept in a hot air oven at 100 °C. Thus dried material is grinded to acquire nano composite material.

$$Talc + H_2O + Fe^{2+}(aq) + 2Fe^{3+}{}_{(aq)} \xrightarrow{\text{stirring}} [Talc/Fe^{3+}\text{-}Fe^{2+}]$$

$$[Talc/Fe^{3+}\text{-}Fe^{2+}] + 8\,OH^{-}(aq) \longrightarrow [Talc/Fe_3O_4]\,(S) + 4H_2O(L)$$

2.4 Preparation of Methylene Blue (Mb) Stock Solution

About 125mg of methylene blue dye was weighed and transferred into 250 ml volumetric flask. Distilled water was added up to mark in 250 ml volumetric flask to prepare 500ppm solution of methylene blue solution. 1ml of solution was taken from 500ppm stock solution in 100ml volumetric flask and added water up to mark to make 5ppm solution. Similarly different concentration of 10, 15, 20 and 25 ppm solutions were prepared.

2.5 Photocatalytic Degradation Studies

Solution of 5ppm concentration was prepared by taking 1ml of MB dye into 100 ml volumetric flask and added water up to the mark. Solution of Nano composite dosage 50mg and 5ppm concentration were set to pH of 1,3,5,7 & 9 by adding HCl and NaOH drop wise to get desired values of pH. The prepared solution was placed into tubes in photocatalytic reactor under UV-Vis light source. 2ml of sample was taken for every 30 min upto 150 min. The bubbling action was done internally by reactor (for proper mixing). Degradation results were studied at different pH (1, 3, 5, 7 and 9) with 5ppm concentration and dosage of 50mg for 150 min contact time. Solutions of different concentration 5, 10, 15, 20 & 25 ppm were taken at pH 9 and 50 mg Nanocomposite dosage for degradation studies. At 5 ppm concentration highest degradation of 65% occurred at 150 min contact time. Different nano-composite dosage of 10, 30, 50, 70 and 100mg were used for degradation of dye at 5 ppm and 9 pH. At 30mg dosage highest degradation of 87% occurred with contact time of 150 min. Adsorbent dosage, effect of pH, contact time & concentration were used as the independent variables and their effects on degradation of dye were investigated elaborately.

3. RESULTS AND DISCUSION

3.1 X-Ray Powder Diffraction (XRD)

The Fig. 1 represents synthesized nano iron oxide was identified as Talc/Fe_3O_4 magnetite was compared JCPDS (01-080-0315). Patterns of composite (Talc/Fe_3O_4), demonstrates crystalline nature with size 49.1nm using Debye-Scherrer [13-15].

$Dp=0.94\lambda/\beta \cos\theta$,

Where, Dp = Particle size (in nm), β = Line broadening (in degrees)= $3.0529*10^{-3}$, θ = Bragg angle (in degrees)=14.3122, λ = X-ray wavelength = $1.54*10^{-1o}$ nm.

3.2 Fourier Transform Infrared Spectroscopy

Sample was mixed with KBr (Potassium Bromide), which is grinded into uniform fine powder and pressed into pellets to obtain FT-IR spectra. From Fig.2 peak at 3418 and 1630 cm^{-1} designates the water absorption and presence of hydroxyl group respectively. Vibrational bands in region 400-1000cm^{-1} owing to Si-O and strong bonds at 1014 and 1349 cm^{-1} was associated with stretching vibration of Si-O, The bond at 663 cm^{-1} was also assigned to stretching vibration of Si-O. The band at 410–301cm^{-1} was assigned to Si-O-Si bending vibration.

3.3 Scanning Electron Microscope (SEM) and EDAX Analysis

The external surface layer of Talc/Fe_3O_4 nano composites were shown in Fig.3 with high magnification of 20, 10 and 5 µm. Images reveals fine-grain, sheet-like microcrystalline talc crystals are associated with acicular actinolite and curved and flake-like morphology.

Energy-dispersive X-ray spectroscopy is an analytical technique used for the elemental analysis or chemical characterization of a sample. The energy-dispersive X-ray spectroscopy results are shown in Fig. 4. It witnesses the presence of elements C, O, and Fe, Si, Mg, N are present.

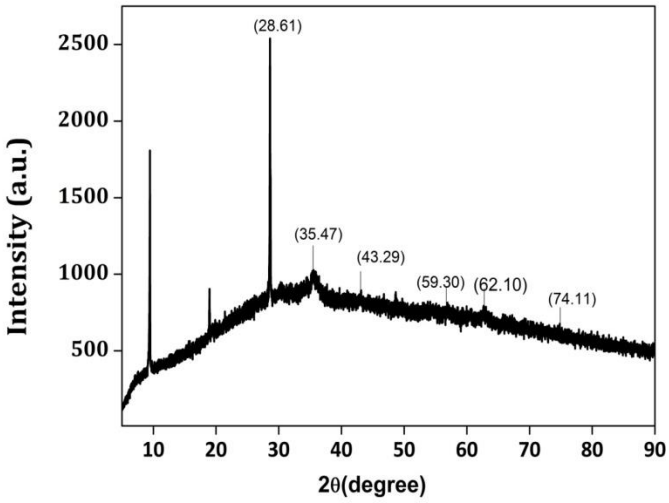

Fig 1: XRD pattern of Talc/Fe_3O_4nano composite material (JCPDS code 01-080-0315)

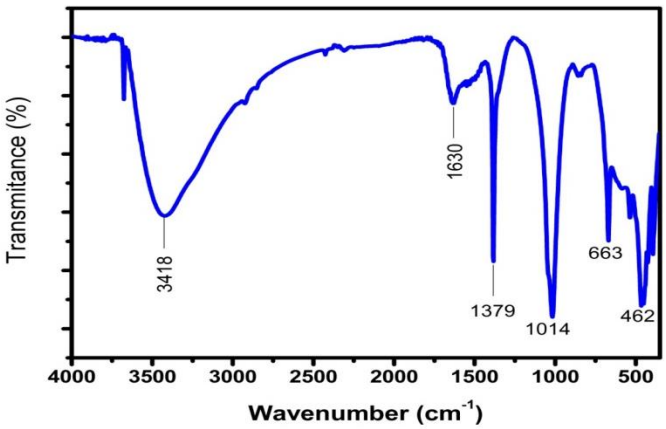

Fig 2: FTIR pattern of Talc/Fe_3O_4nano composite material

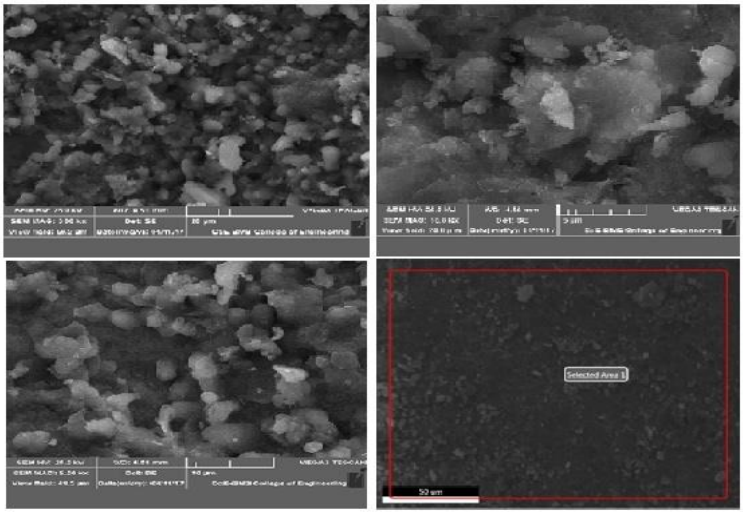

Fig 3: Scanning electron microscopy micrographs of the Talc/Fe_3O_4nanocomposites at different magnifications

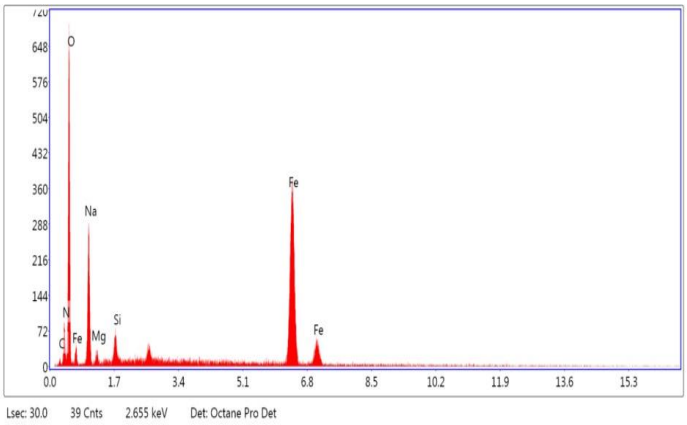

Fig 4: EDX pattern of Talc/Fe_3O_4 nano composites

3.4 Mechanism of Photo Catalytic Degradation of MB Dye

The photocatalytic degradation is one of the most significant processes to remove hazard nature of organic dyes from the textile effluent. The semiconductor photo-catalysts employ light energy to generate electron/hole (e^-/h^+) pairs on the surface of photo-catalyst. These pairs carry out oxidation and reduction processes, which are by and large renders the reactive oxygen leads to active species for the decomposition of contaminants. Hydrogen peroxide (H_2O_2) was employed to enhance the production of reactive oxygen species, extra oxidants [16]. It is known through literature that the pH of the solution affects the photo-degradation of dyes. Therefore degradation studies were done in the pH range of 1 to 9. The photo-degradation of dye too enhances with the increase in pH and at pH 9 most prominent degradation observed. Further increase in pH causes the reaction rate to decrease. This increase in degradation is because of the interaction between the cationic dye molecules and hydroxyl ions with a increase in pH. Any increase above pH 9 causes the degradation reduction and this may be due to the electrostatic repulsion. The dye gets adsorbed easily on talc/ Fe_3O_4 surface only in low pH range because of the electrostatic forces of attraction [16]. Best results of degradation were observed at concentration of 5 ppm, dosage of 30mg, and pH9 as shown in graph Fig.6 and Fig.7. With an increase in nano composite dose the agglomeration of particles takes place beyond the optimized value [16-17].Studies carried out on the dye concentration revealed that the degradation reduced with increase in concentration. As seen in Fig.7, the degradation reduced with MB dye concentration increase above 5ppm after 150 minute irradiation exposure. More active sites are made available at low concentrations but at higher concentrations molecules of MB dye get adsorbed causing hindrance in catalyst adsorption on the surface [17].

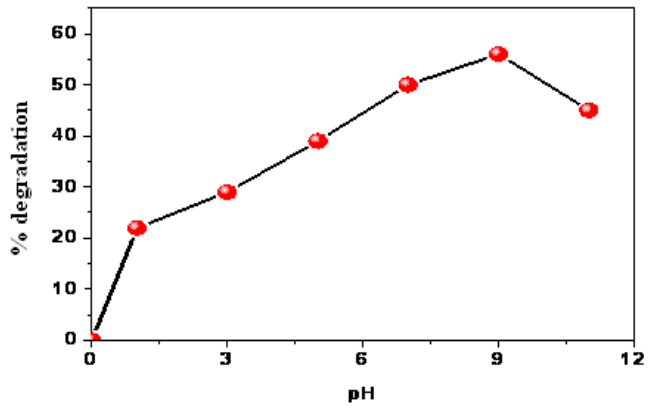

Fig 5: % degradation v/s different pH, at 50 mg catalytic dosage, 5ppm concentration, at 150 min.

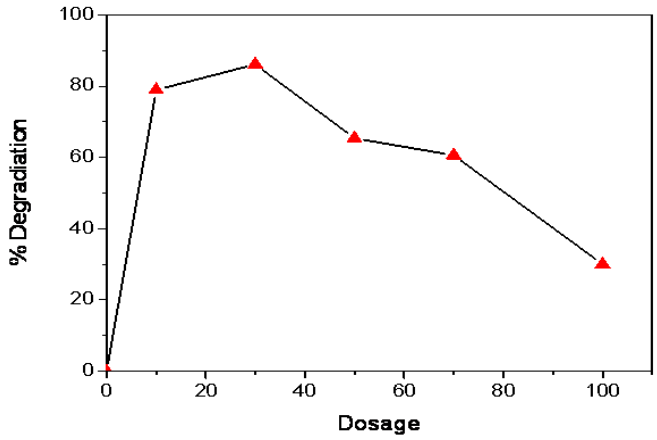

Fig 6 (%) degradation v/s different dosage, at pH 9, 5ppm concentration, at 150 min.

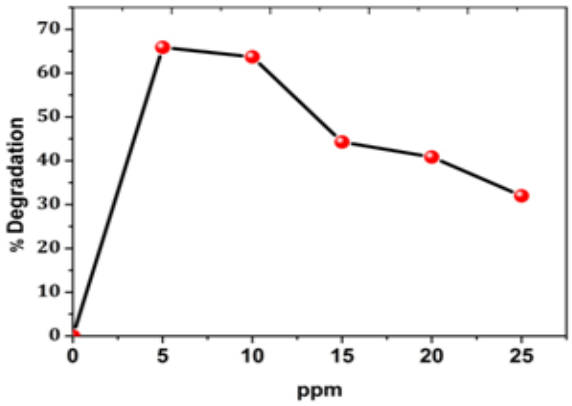

Fig 7 (%) degradation v/s different concentration, at pH9, at 50 mg dosage , at 150 min

4. CONCLUSION

The XRD results of synthesized nano composite material show crystalline size of 49.11nm using Debay Scherrer's equation. SEM analysis demonstrates circular in shape of samples. The presence of Fe, C, Si, Mg and O in nano composite material was confirmed by EDX. Talc/Fe_3O_4nanocomposite was used as an efficient photo catalyst for the degradation of MB under UV-vis light. The catalyst showed high degradation in basic medium for the absolute photocatalytic

degradation at 9 pH. Degradation decreases with time above 150min of UV-vis light in basic medium. It was observed that at pH 9, complete degradation took place in 150 min exposure to UV light at 30mg dosage and at 5 ppm concentration.87% of degradation was recorded. The increase of Talc/Fe_3O_4nanocomposite dosage beyond the optimum of 30mg results in the agglomeration of NC particles, hence more NC particle surface become unavailable for photo absorption, and degradation rate of NC particle decreases.

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